Continuous Reflectivity Measurements for Growth Rate and Emissivity-Corrected Pyrometry in MBE Systems

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Introduction and Motivation

Obtaining continuous reflectivity measurements in MBE systems is challenging due to the variation in the reflectivity signals caused by substrate wobble. These variations are exacerbated by the relatively long substrate-to-detector distances in MBE systems (typically greater than 24") compared to MOCVD systems (typically less than 12"). This results in oscillations in both the measured reflectivity and the temperature. With optics configured to mitigate these effects, the kSA Integrated Control for Epitaxy (kSA ICE) instrument provides reflectivity and emissivity-corrected pyrometry (ECP) measurements that can be utilized with MBE for real-time epitaxial material calibration and process control in both continuous and triggered modes.

Instrument Configuration

Like kSA ICE for MOCVD, kSA ICE for MBE is a modular, *in situ* metrology system that combines multiple kSA measurement techniques. The heart of the kSA ICE metrology tool is a real-time, single-wavelength reflectivity measurement used to calculate film thickness, growth rate, and optical constants (formerly performed by kSA RateRat) combined with an ECP measurement used to accurately determine temperature by correcting for changes in the emissivity of the sample during film growth. By utilizing the virtual interface method developed at Sandia National Laboratory and licensed by k-Space, optical constants of the growing film can be extracted by restarting the fitting routine with each new layer and treating the underlying film stack as a "virtual" substrate. [1]

The kSA ICE ECP system uses a photodiode to measure the intensity of the blackbody radiation emitted from a sample at a specified wavelength (typically 940 nm). This intensity is then used to calculate the uncorrected pyrometer temperature. During the deposition of semitransparent films, the uncorrected pyrometer temperature may appear to change or oscillate due to a change in surface emissivity resulting from thin film interface and/or surface roughening. This apparent change in temperature is not necessarily "real" and can be corrected if the change in emissivity is known. The emissivity of a surface can be calculated by subtracting the measured reflectivity from unity, provided the sample is absorbing at the wavelength in question.[2] The kSA ICE ECP system measures

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[1] Breiland, W. G., & Killeen, K. P. (1995). A virtual interface method for extracting growth rates and high temperature optical constants from thin semiconductor films using in situ normal incidence reflectance. *Journal of Applied Physics,78*(11), 6726-6736.

[2] Breiland, W. G. (2003). Reflectance-Correcting Pyrometry in Thin Film Deposition Applications. *SAND Report*. Retrieved from https://prod-ng.sandia.gov/techlib-noauth/access-control.cgi/2003/031868.pdf. SAND2003-1868.

surface reflectivity in real-time using a photodiode and an integrated light source, in this case a 940 nm LED. This reflectivity value is used to calculate the emissivity, which in turn is used to obtain the emissivity corrected temperature. The kSA ICE ECP measurement uses alternating light-on/light-off photodiode measurements to measure the reflectivity and the uncorrected pyrometer temperature, respectively. In continuous mode, the user can define the measurement cycle time. In triggered mode, the application synchronizes the measurement to sample rotation and updates the results upon every other rotation. The user can calibrate the pyrometer temperature using a standard calibration technique, or they can opt to integrate their system with a kSA BandiT system for calibration via Band Edge Thermometry or Blackbody Temperature Fitting.[3] The reflectivity is calibrated at the start of the growth using the known reflectivity of the bare wafer at the wavelength of interest.

Depending on the customer's application, measurement needs, and/or safety requirements, the kSA ICE can be configured with a variety of different wavelengths and light sources, including lasers or LEDs. The data presented below is from a kSA ICE instrument with a 532 nm fiber-coupled LED for reflectivity measurements, and a 940 nm fiber-coupled LED for reflectivity and ECP measurements. The optics were located one meter from the sample. To accommodate such large working distances, the kSA ICE optics utilized a 2" objective lens in conjunction with photodetectors mounted directly on the optical head to collect a larger angular range of reflected light from the substrate. An x-y translation stage was used to enable fine control to optimize the measurement position. To further minimize the effect of the long path length, kSA ICE measurements can be synchronized to substrate rotation to remove the wobble-induced oscillations in the reflectivity measurement.

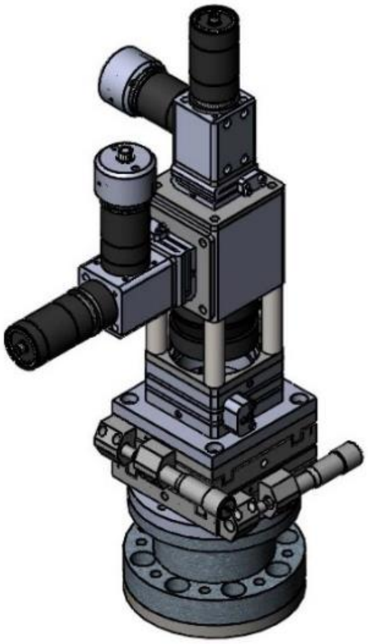

Figure 1: *A kSA ICE-ECP-MBE instrument configured with LED-based 940nm and 532nm reflectivity, and emissivity corrected pyrometry. The unit includes x-y translation and tilt capability.*

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[3] BandiT Pyrometry for Temperature Determination, https://www.k-space.com/wp-content/uploads/BandiTPyrometry.pdf

Measurement and Data

Figure 2 shows the simultaneous measurement of the surface reflectivity at 532 nm and 940 nm during growth of AlAs and GaAs films on a GaAs substrate. For a deposition with constant growth rate, the oscillation period is proportional to $\lambda/2n$. As such, the oscillation period of the 532 nm reflectivity data is shorter than that of the 940 nm reflectivity data, as seen in Figure 2. A shorter oscillation period allows the fit to converge earlier in the growth. As a result, the shorter wavelength 532 nm reflectivity data allows the calculation of film thickness earlier in the growth and allows characteristically thinner layers to be measured (See Figure 3.) While this can be an advantage in some cases, in the case of GaAs film growth shown below, the absorption of 532 nm light is much stronger than that of 940 nm light, causing the 532 reflectivity to damp out before the time the target film thickness reached, approximately 5 periods (~350 nm). This limits the use of 532 nm reflectivity to monitor film thicknesses under 350 nm. Conversely, 940 nm reflectivity continues to produce strong oscillations beyond 350 nm.

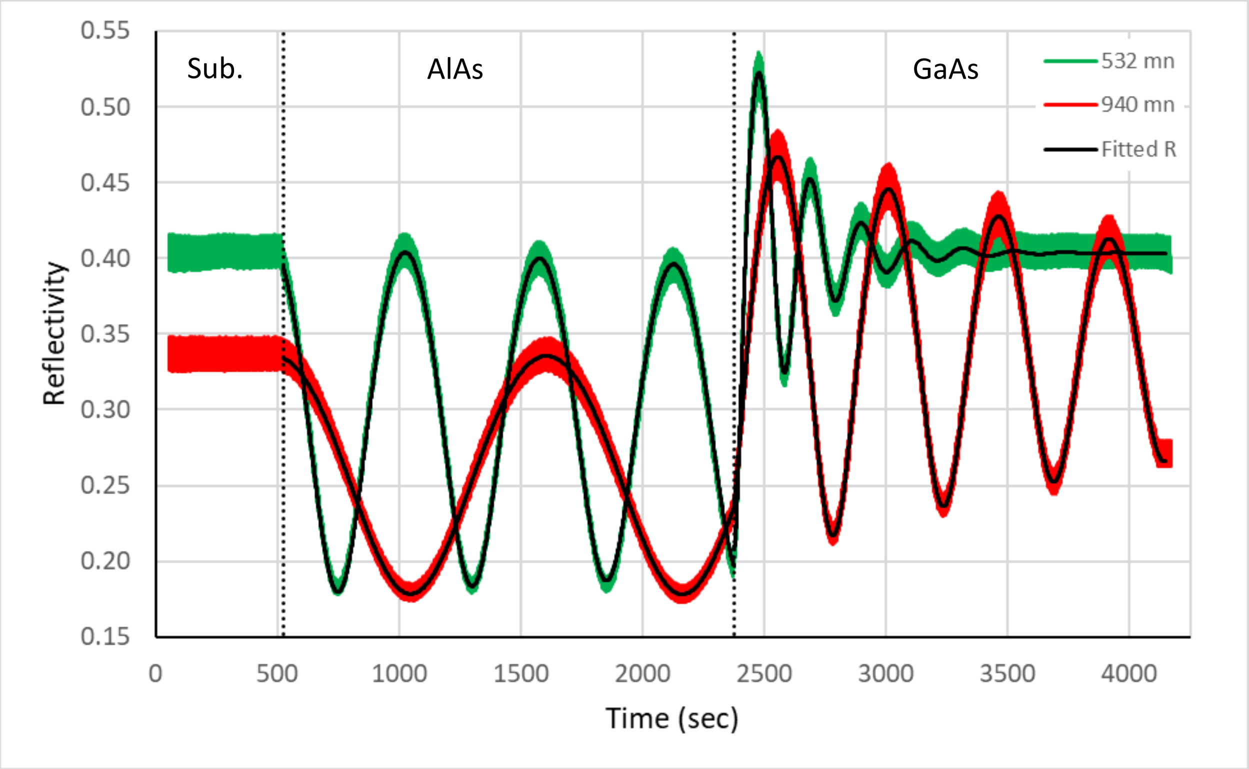

Figure 2: *532 and 940 nm reflectivity during growth of 250 nm of AlAs followed by 500 nm of GaAs on a GaAs substrate, and the corresponding virtual interface reflectivity fits for each layer and wavelength.*

To illustrate the wavelength dependence of the growth rate (*G*) and optical constants (*n, k)* during the AlAs growth shown in Figure 2, Figure 3 shows the real-time fitting results for each value; the measured reflectivity is also shown for reference. Note that the fit for *n* and *G* converges before the thickness reaches half an oscillation period and becomes quite stable by the time a full period is reached. The *k* value is more challenging to determine, as it is tied to the damping of the envelope of the oscillations. Therefore, it tends to take longer to converge, and can exhibit relatively small oscillations, as seen in these results.

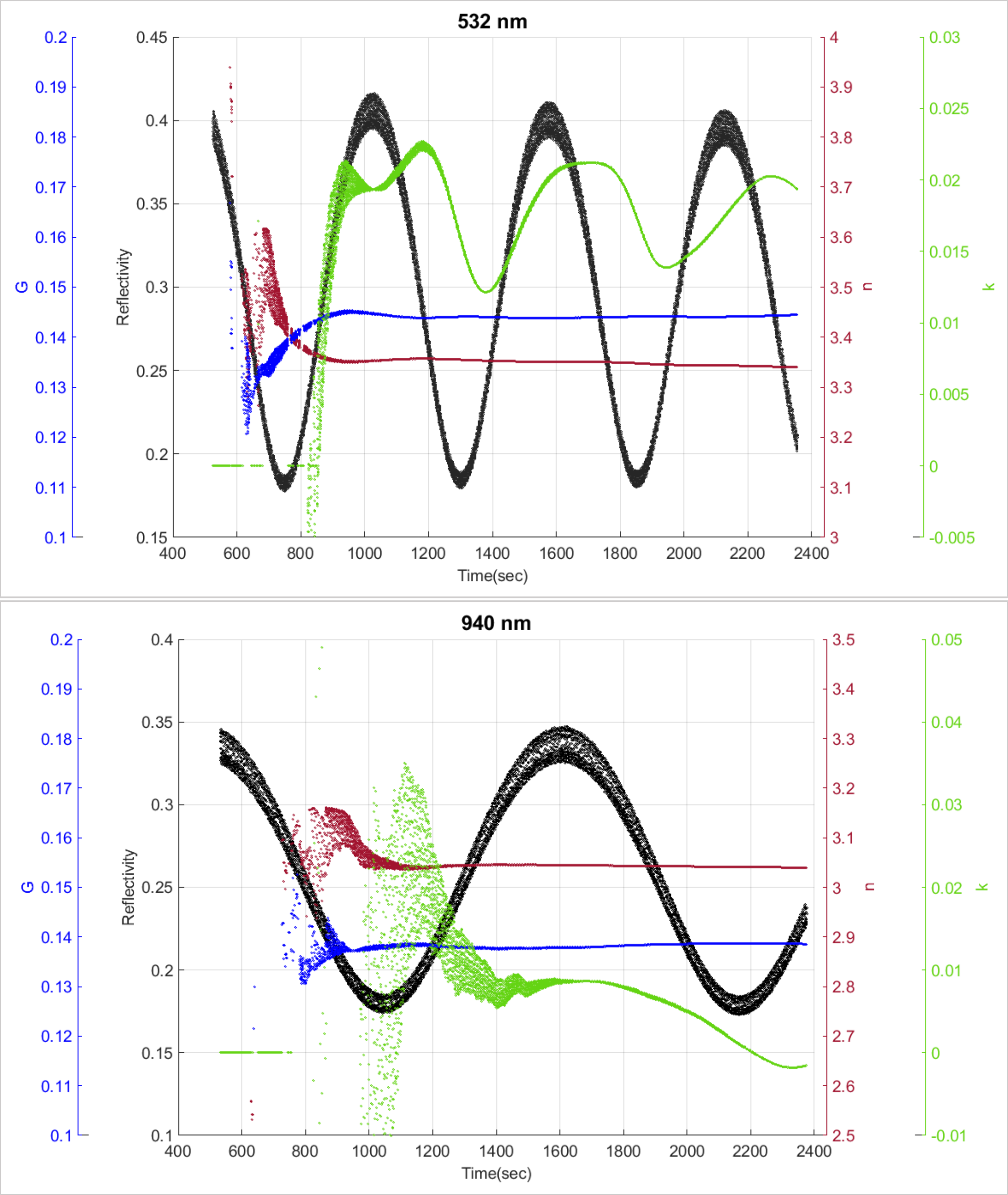

Figure 3*: Real-time fitting results for the growth rate and optical constants during the AlAs growth shown in Figure 2. The measured reflectivity is also shown for reference.*

The reflectivity oscillations in Figure 2 and 3 indicate that signal scatter is present in both the 532 and 940 nm reflectivity data. Figure 4 shows a detailed view of the 532 nm reflectivity signal during the GaAs growth. The short-period oscillations in the reflectivity data result from inherent wobble of the MBE wafer platen and are approximately ± 0.015 in magnitude. Despite these oscillations, the kSA ICE tool can effectively fit through this noise, allowing a precise determination of the film's optical constants. The fitted values for *n*, *k*, and *G,* as determined from 532 nm and 940 nm reflectivity data from AlAs and GaAs films, are displayed in Table 1. Both measurements resulted in calculated growth rates within 5% of each other and the calculated values for *n* and *k* were found to exhibit wavelength dependence as expected.

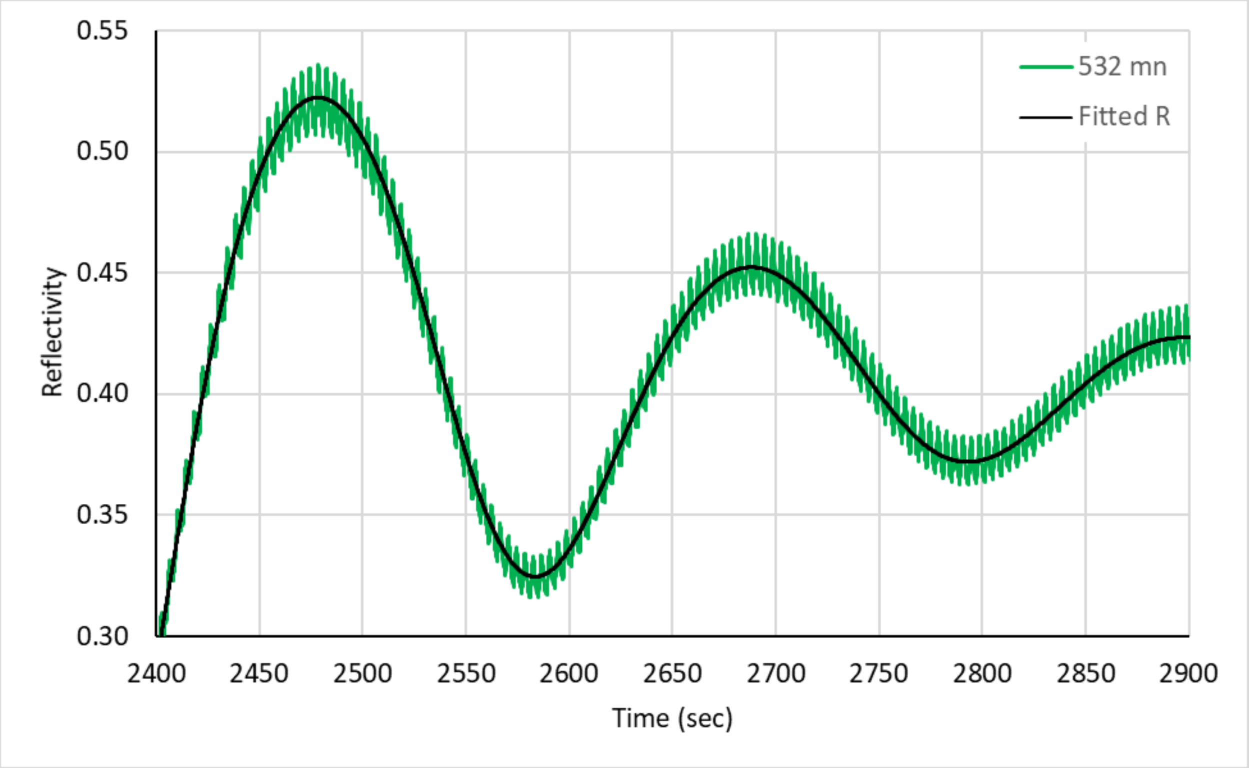

Figure 4: *Detailed view of the 532 nm reflectivity during the GaAs growth. Note the excellent fit despite the strong absorption and the wobble-induced short-period oscillations.*

	AlAs			GaAs		
532 nm	*n* = 3.340	*k* = 0.019	*G* = 0.144 nm/s	*n* = 4.385	*k* = 0.635	*G* = 0.290 nm/s
940 nm	*n* = 3.040	*k* = 0.000	*G* = 0.139 nm/s	*n* = 3.757	*k* = 0.107	*G* = 0.276 nm/s

Table 1: *Fit results from the AlAs and GaAs film growth shown in Figure 2.*

In addition to measuring *n*, *k*, and *G*, kSA ICE can be used to measure the sample temperature. Figure 5 shows the corrected and uncorrected pyrometer temperatures from the AlAs and GaAs growth shown in Figure 2. As the AlAs is deposited, the uncorrected pyrometer temperature starts to increase, while the ECP temperature actually decreases from 590°C to 578°C. Later in the AlAs layer growth, the operator adjusts the heater set point to achieve an ECP temperature of 590°C prior to the growth of the GaAs layer. Note the large oscillations in the uncorrected pyrometer temperature of ± 5°C seen during the GaAs layer growth. If one examines the detailed view in Figure 6, it becomes apparent that the short-period oscillations in the corrected temperature are caused by the wobble-induced reflectivity variation; they are effectively removed by real-time smoothing. Also, note that the ECP temperature oscillations are 180° out of phase with the reflectivity oscillations in the 940 nm signal. During growth of the GaAs layer, the heater is manually adjusted by reading the ECP temperature and trying to maintain a growth temperature of 590 °C. Once the heater set point stabilized, the smoothed ECP temperature settled to within ± 0.5°C, whereas the pyrometer still oscillated by ± 5°C during growth. Clearly, the emissivity corrected pyrometer temperature provides a superior means of temperature control when compared to the uncorrected temperature.

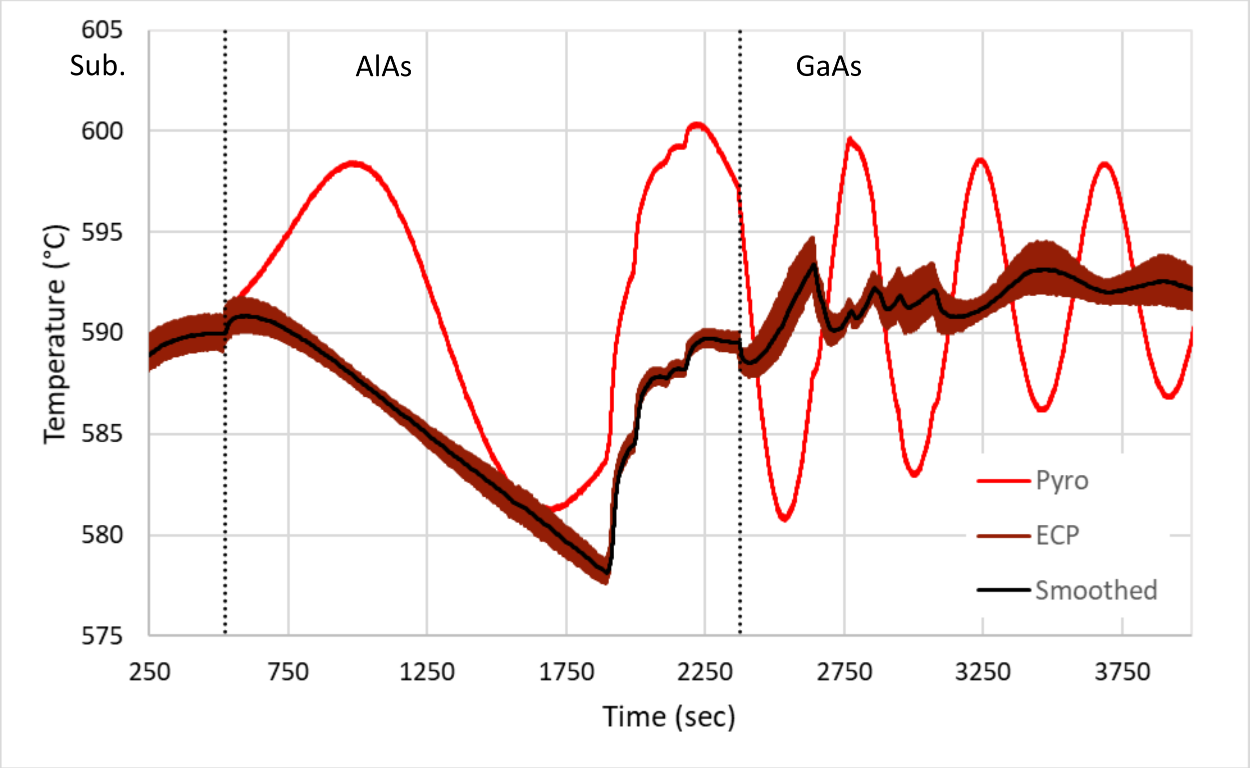

Figure 5: *Uncorrected pyrometry and corrected ECP temperatures from the AlAs and GaAs growth.*

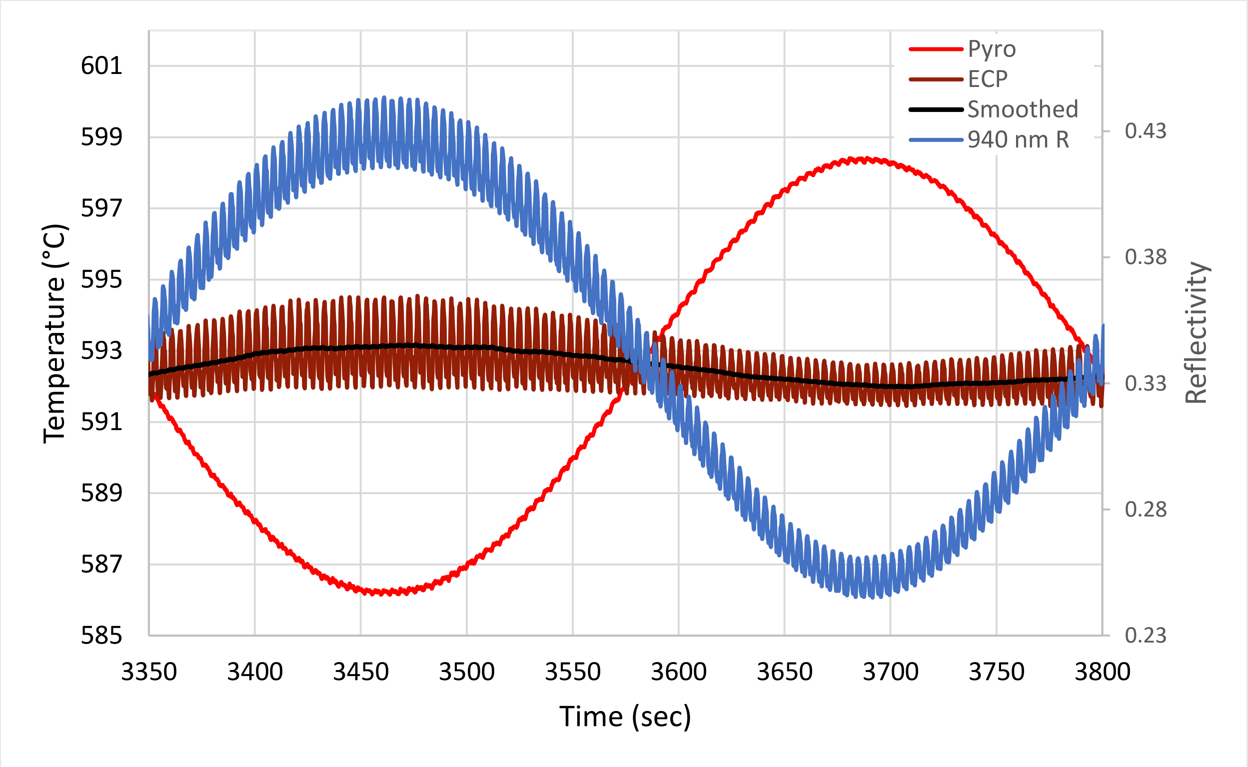

Figure 6: *Detailed view of a single reflectivity oscillation period during the GaAs film growth. Note that the variation in the 940 nm reflectivity induced by substrate wobble leads to short-period oscillations in the corrected ECP temperature. These are effectively removed by real-time smoothing.*

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